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A BRIEF GUIDE TO UF6 HANDLING

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OAK RIDGE GASEOUS DIFFUSION PLANT

Operated by

UNION CARBIDE NUCLEAR COMPANY
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and
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ABSTRACT

Techniques used in the transfer of UF_6 to and from shipping containers are outlined. The physical and chemical properties of UF_6 are also included.

A BRIEF GUIDE TO UF_6 HANDLING

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A BRIEF GUIDE TO UF_6 HANDLING

INTRODUCTION

During the early stages of industrial participation in the program for peaceful use of atomic energy, it is anticipated that UF_6 will be largely supplied to industry by AEC-owned facilities. Also, it has been established that AEC-owned cylinders will normally be supplied for interplant transfers of UF_6, particularly where the origin or destination is an AEC facility.

While it is hoped that this guide will be helpful to the Civilian Licensee in studying the problems associated with UF_6 processing, it was prepared primarily to provide a basis for a common understanding between shipper and receiver. Such pertinent matters as AEC cylinder inspection, cleaning, repair, and filling and emptying techniques are covered. Although available elsewhere, some general information on the properties of UF_6 was compiled for convenient reference.

The methods outlined have proven satisfactory over a period of years and are typical of the techniques which will be used, where applicable, by the AEC Contractor in preparing either empty or full UF_6 cylinders for shipment to industrial participants. Similar handling methods, if adopted by industrial participants, should result in an entirely satisfactory shipper-receiver relationship on the points covered.

PHYSICAL AND CHEMICAL PROPERTIES OF UF_6

Sublimation Point (14.7 psia.)	133.5° F.
Triple Point	22 psia., 147.3° F.
Density, Solid (68° F.)	292 lb./ft.^{3}
Liquid (147.3° F.)	229 lb./ft.^{3}
Liquid (300° F.)	192 lb./ft.^{3}
Heat of Sublimation (147° F.)	58.7 Btu./lb.
Heat of Fusion (147° F.)	23.4 Btu./lb.
Heat of Vaporization (147° F.)	35.3 Btu./lb.
Heat of Solution in Water (77° F.). Heat evolved	90,900 Btu./lb. Mol
Critical Pressure	45.5 atm.
Critical Temperature	446° F.

At room temperature, UF_6 is a white, volatile solid which forms transparent crystals that sublime slowly at atmospheric pressure; at higher pressures they melt to form a clear, colorless liquid of high desnity. Uranium hexafluoride is a highly reactive substance. It reacts chemically with water, ether, and alcohol forming soluble reaction products. It reacts with most organic compounds and many metals. Most saturated halocarbons are of low reactivity or inert to UF_6. It does not react with oxygen, nitrogen, and dry air. It is sufficiently inert to copper, nickel, aluminum, teflon, monel, and aluminum bronze that they can be exposed to UF_6 without excessive corrosion. Low silicon steel is sometimes used where service is mostly at room temperature.

Uranium hexafluoride reacts very readily with H_2O to form UO_2F_2 and HF. A saturated solution of UO_2F_2 in water has the following composition:

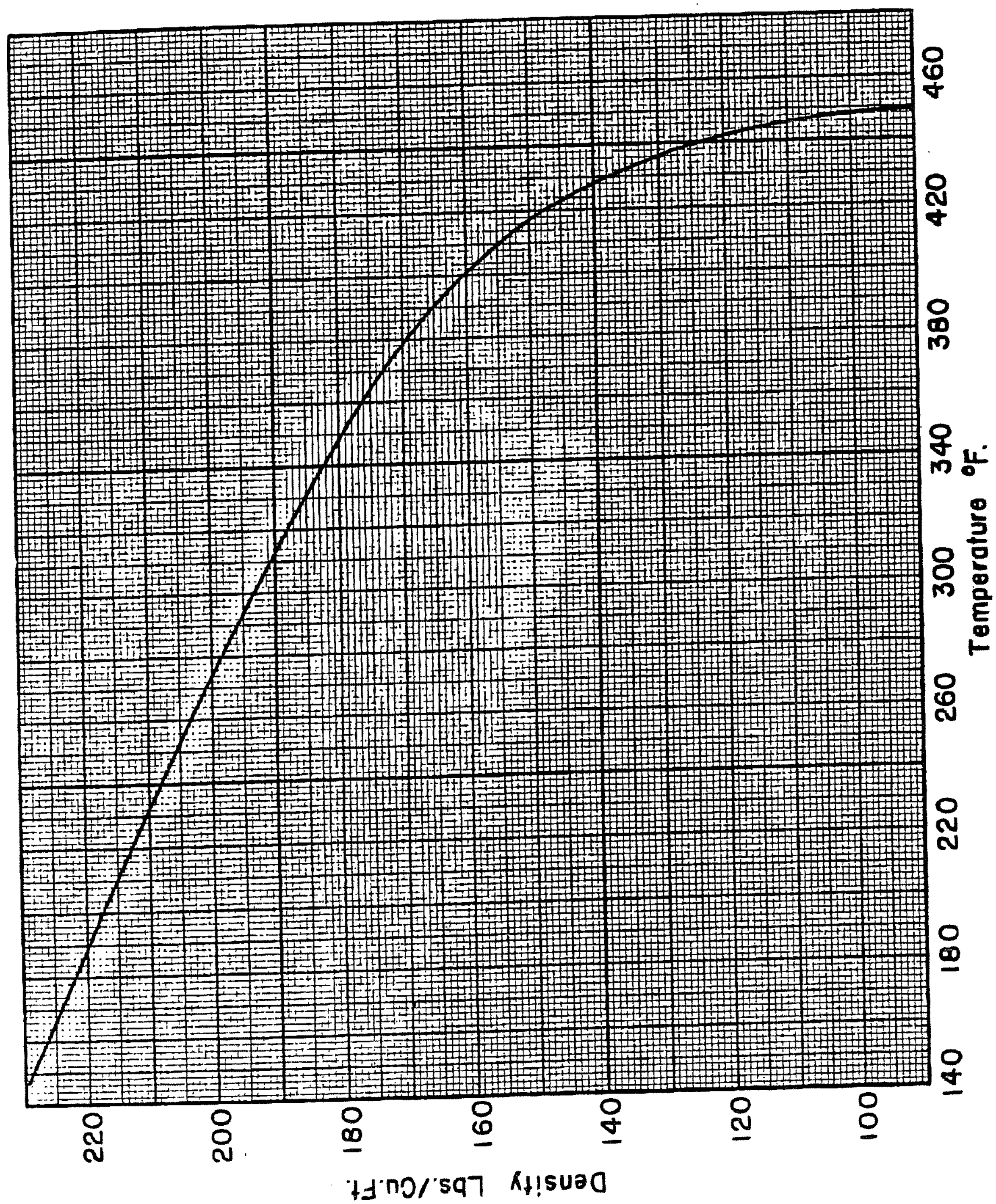
°C.	% UO_2F_2
1	61.4
25	65.6
60	71.0
100	74.1

There is a marked decrease in solubility of UO_2F_2 with increasing concentration of HF. This is illustrated by the following table:

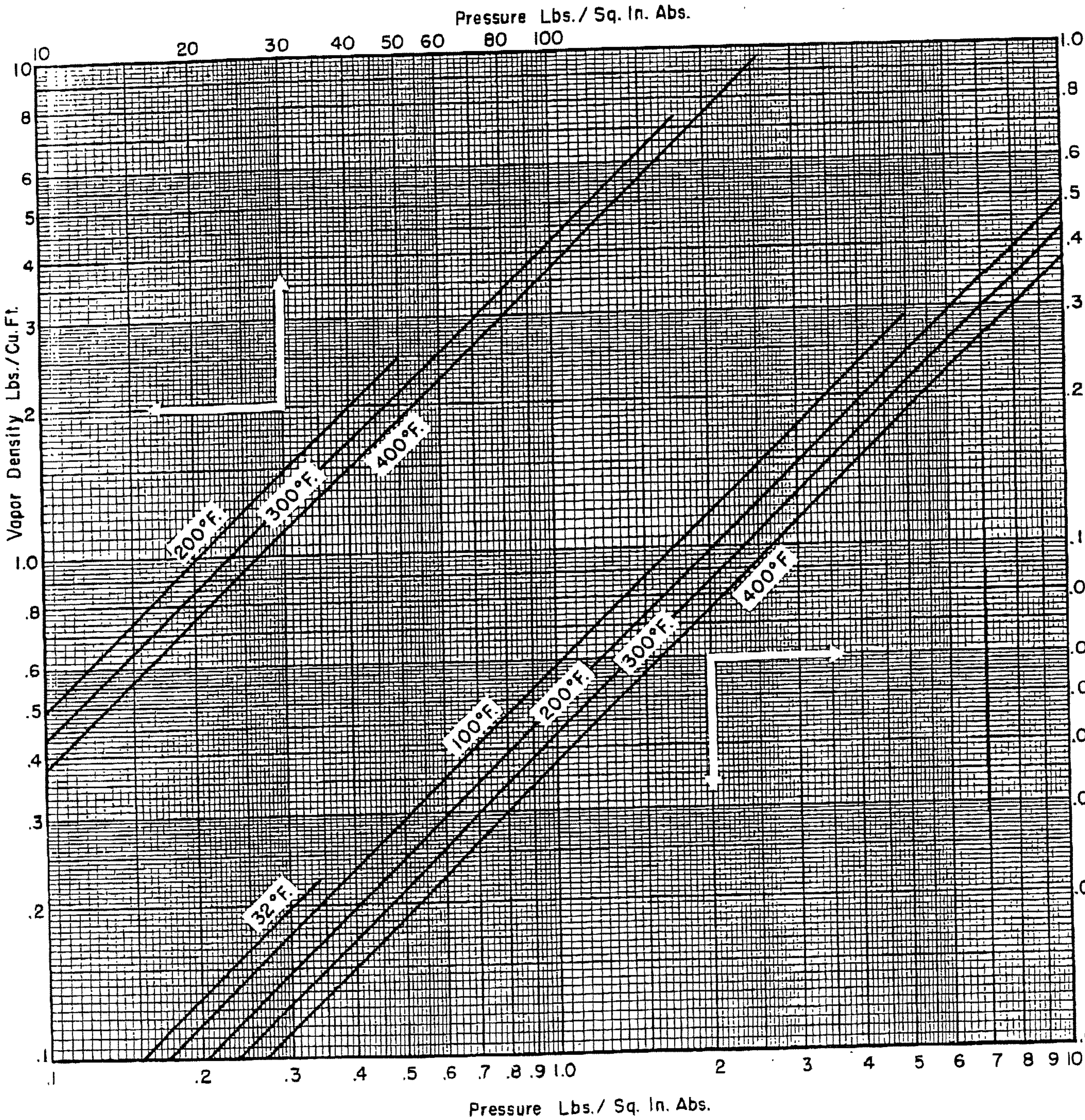
SOLUBILITY OF UO_2F_2 AT 25° C.

HF (%)	UO_2F_2 (%)	H_2O (%)
0.00	65.55	34.45
1.28	47.58	51.14
3.59	39.78	56.63
9.78	32.25	57.59
11.88	31.88	56.24
20.70	22.29	57.01
25.75	18.19	56.01

DENSITY OF LIQUID UF_6



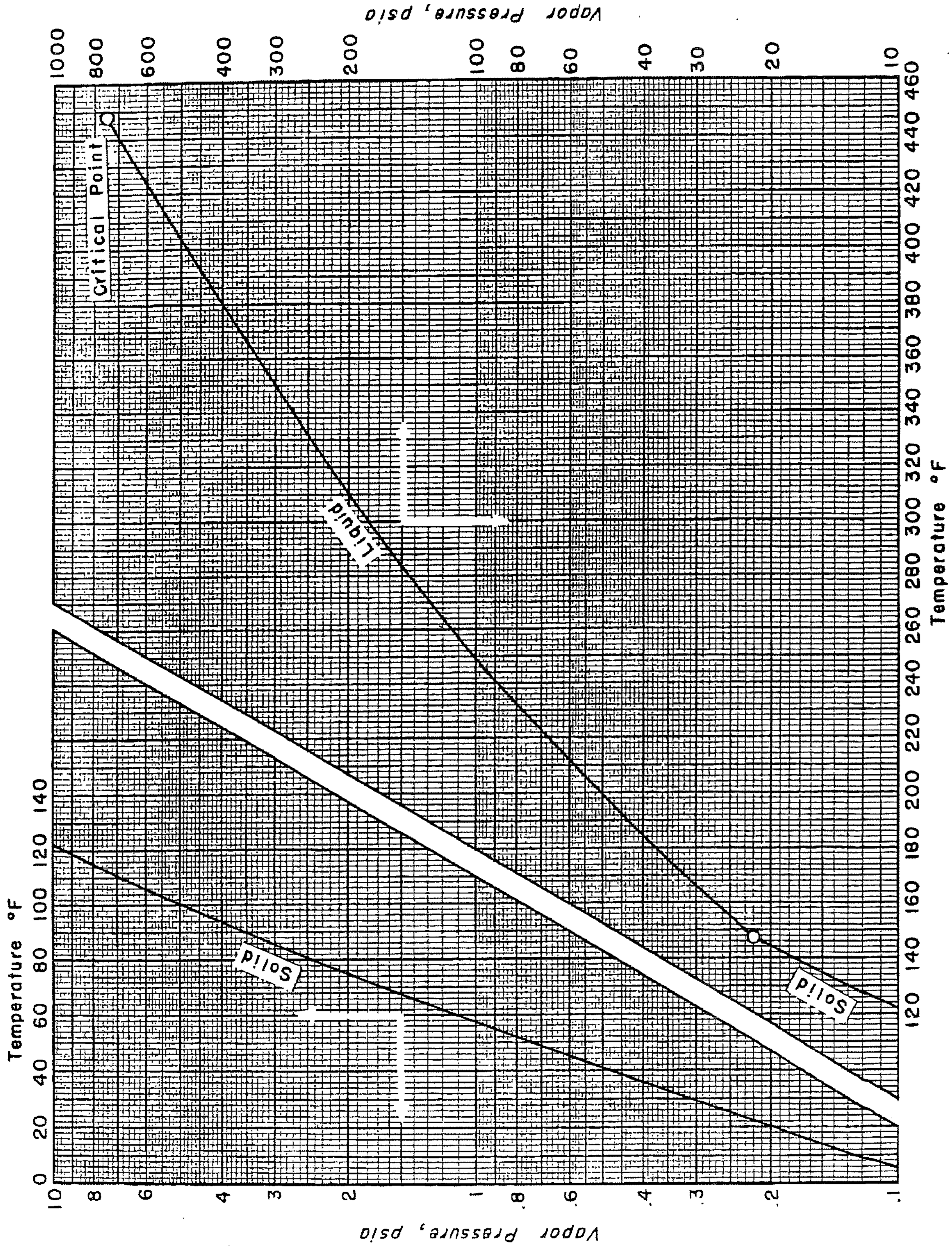
DENSITY OF GASEOUS UF_6

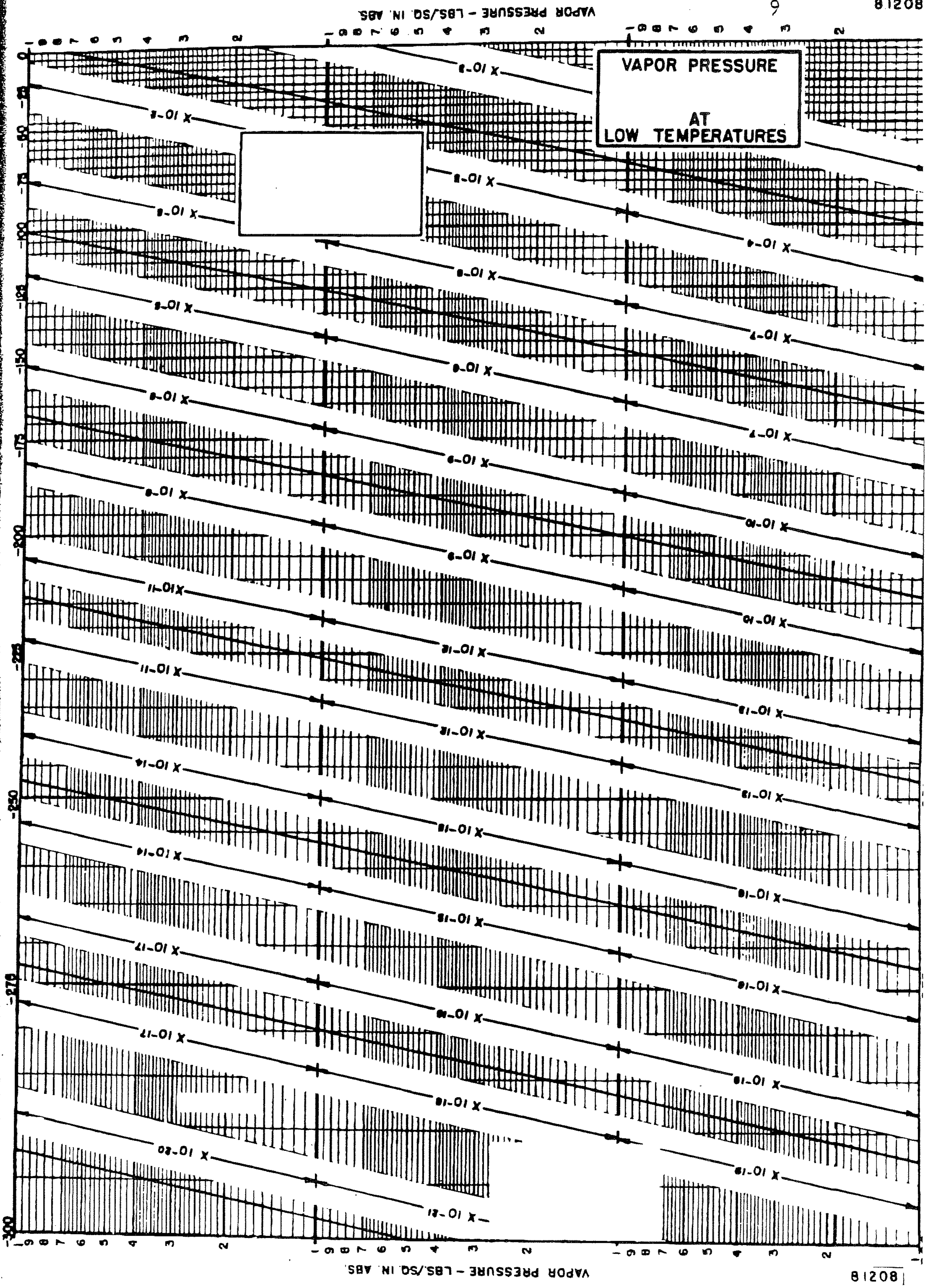


VAPOR PRESSURE

OF

URANIUM HEXAFLUORIDE




VAPOR PRESSURE
AT
LOW TEMPERATURES
VAPOR PRESSURE - LBS/SQ. IN. ABS.
0
-25
-50
-75
-100
-125
-150
-175
-200
-225
-250
-275
-300

HAZARDS

Criticality: Non-critical at normal assay or below. Enhanced material requires special considerations not covered here.

Explosive: Reacts with hydrocarbons and many other organic compounds; under confinement these mixtures produce explosive pressures. Cylinders of UF_6-hydrocarbon oil mixtures have exploded and ruptured the cylinders.

Flammable: Not flammable.

Corrosive: UF_6 and UF_6-hydrolysis products are highly corrosive to most materials, including:

1. Body tissues: causes severe burns.
2. Organic compounds.
3. Many metals.

Radiation: Uranium emits alpha radiation, and the decay products emit beta and gamma rays. Biological effects of radiation are not discussed in this guide.

Toxic:

1. UF_6 reacts with moisture in air forming a white fog consisting of UO_2F_2, which is toxic, and HF, which is highly toxic and corrosive.

2. Internal toxicity of uranium compounds varies with the method by which they reach the inside of the body. Inhalation is far more serious than ingestion. The primary action of uranium chemical poisoning is on the kidney.

SAFETY CONSIDERATIONS

General: Avoid physical contact with or inhalation and ingestion of UF_6, or its reaction products.

Design:

1. Use closed system or container.

2. Adequate means for control of possible accidental releases, including necessary hooding and exhaust devices, and treatment of floor and work surfaces for ease of removal.

3. Adequate control of vent gases.

4. Avoid rupture of container due to overheating or overfilling.

5. Use clean equipment to avoid explosive reactions.

STORAGE OF URANIUM HEXAFLUORIDE

Aside from critical mass considerations, the storage of UF_6 poses few problems. Requisites for satisfactory storage are as follows:

1. Approved containers, properly spaced and secured. Cap on valve outlet port.
2. Avoidance of mechanical damage to containers or container valves.
3. Cool, well-ventilated, isolated locations.
4. Protection from fire or excessive heat.

CYLINDERS AND CYLINDER SERVICING

Atomic Energy Commission cylinders available for licensee service are shown in Figure 1, page 13. Owing to criticality considerations, the size of the cylinder must be decreased as the degree of U-235 enrichment increases; e.g., it is anticipated that UF_6 shipments will be limited almost exclusively to 5-inch cylinders for isotopic assays in excess of about 3 weight percent U-235. The maximum allowable cylinder loadings and a discussion of critical mass problems in general are considered beyond the scope of this report. It is expected that this information will be made available by the Commission at a later date.

Cleaning and Inspection

Empty cylinders received from AEC contractors should need no cleaning. A visual inspection should be made on all cylinders for cracked valve nut or body or damaged valve stem or threads.

Pressure Testing

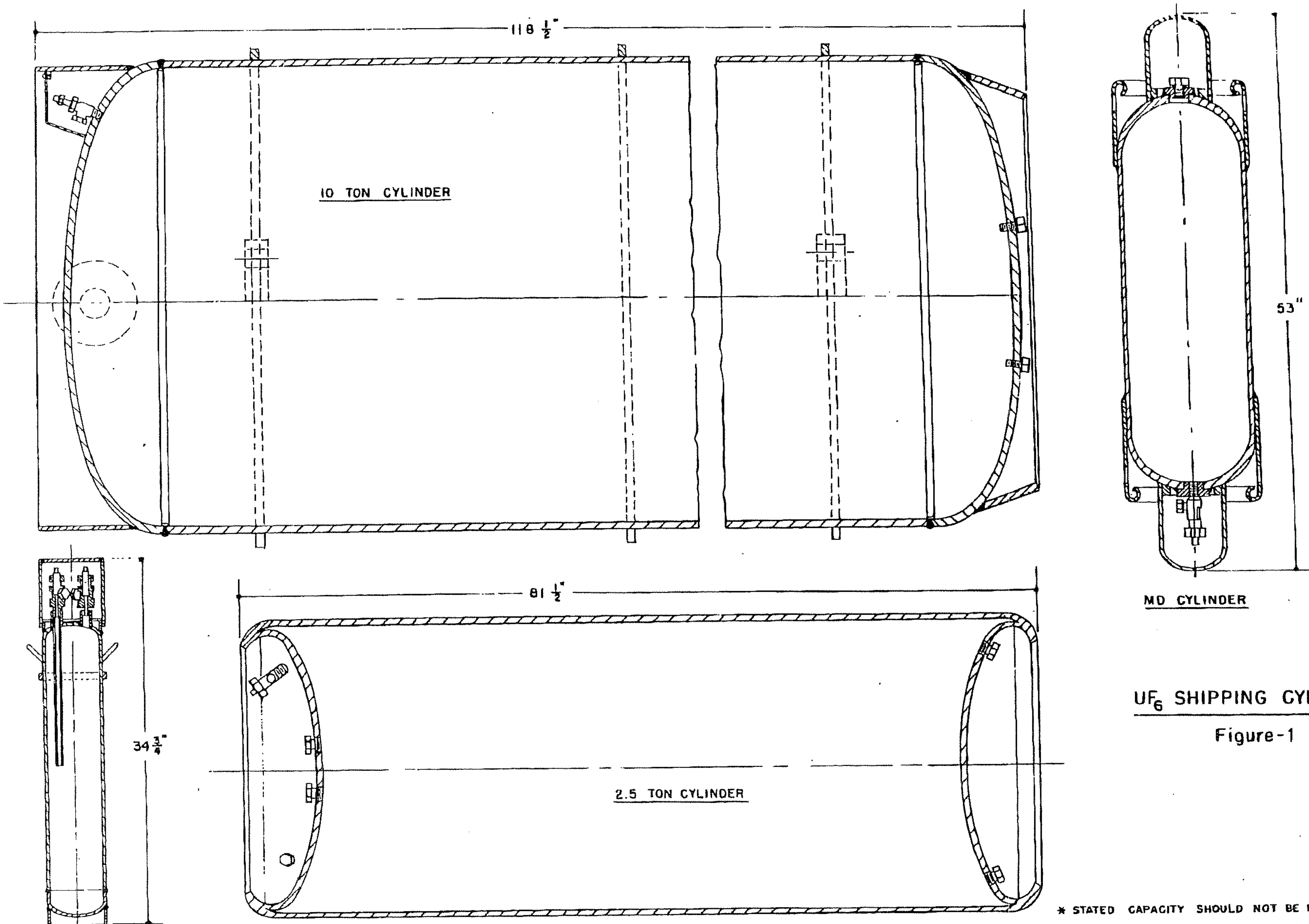
Cylinders are hydrostatically tested at 400 psig. by AEC contractors. Freon 113 is used as the hydraulic fluid to facilitate drying the cylinder after testing.

Leak Testing

An empty leaking cylinder can be detected when it is connected to the manifold and leak rated and the leak located by pressuring to 5 psig. and soap testing. A leak in a cylinder containing UF_6 will be indicated by a white fog issuing from the leak even at room temperature. In time, a small leak may build up yellow or light green deposit at the leak.

Valves and Valve Repair

Drawings of the two valves are shown in Figures 2 and 3, pages 14 and 15. Bridgeport alloy E 3712 (aluminum silicon bronze) has been found a most serviceable material for cylinder valve bodies. The stems are of monel. Monel valve bodies with monel stems have been used, but these are not recommended because of the difficulty experienced with valve seat leakage. Valves are screwed and silver soldered into 5-inch cylinders and screwed into all other UF_6 cylinders. A valve packing leak may usually be stopped by tightening the packing. If this fails to stop the leak, the cylinder can be frozen down with dry ice and the packing replaced. Chevron packing originally supplied has been replaced with flat teflon packing. If a leak through the valve seat cannot be stopped with reasonable torque, it may be possible to feed the cylinder out before replacing the valve. If unsuccessful, the cylinder may be frozen down and the valve changed.



UF$_6$ SHIPPING CYLINDERS

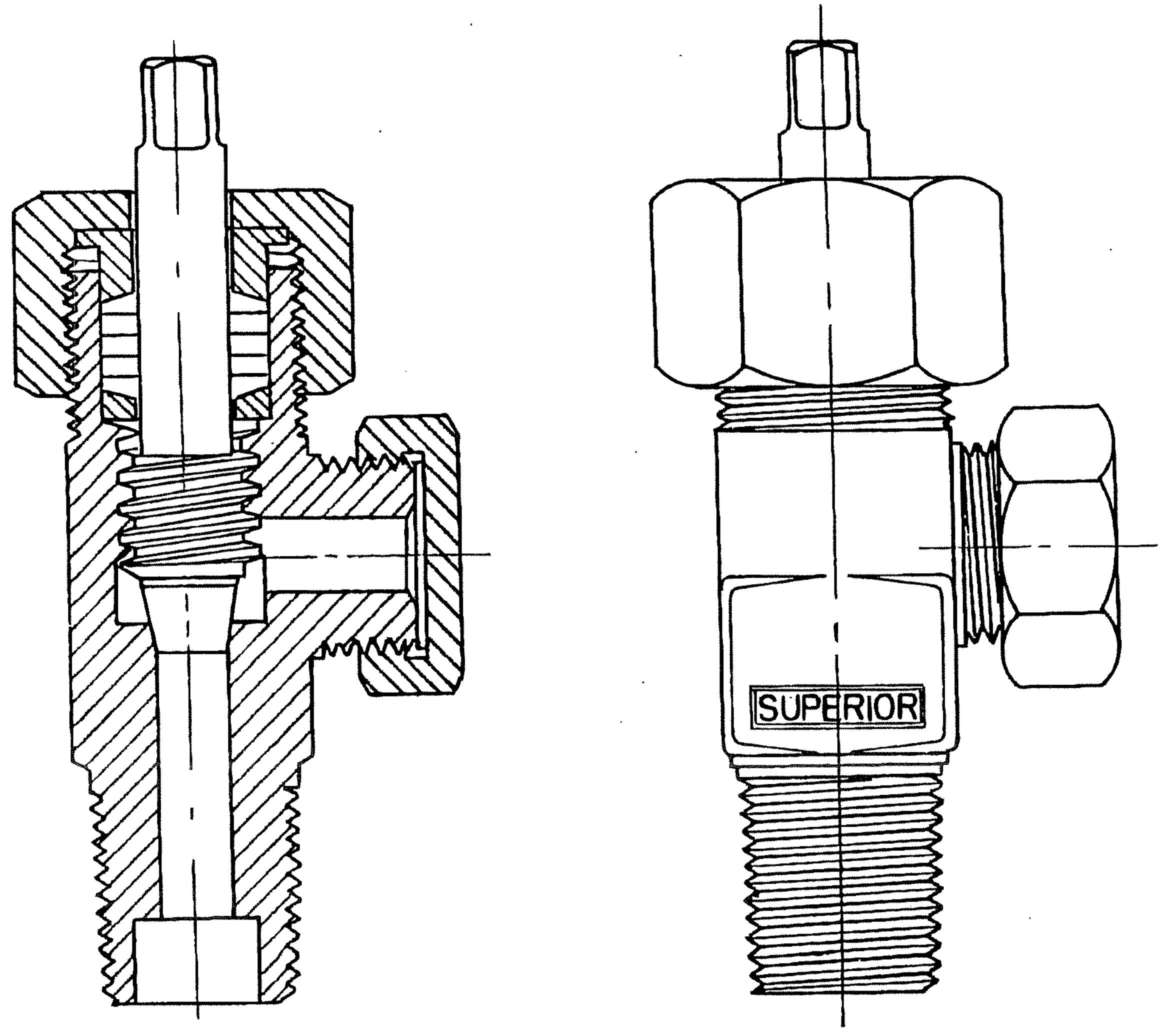
Figure-1

* STATED CAPACITY SHOULD NOT BE EXCEEDED

TYPE	DIMENSIONS	MATERIAL	EMPTY WEIGHT	CAPACITY *	TYPE VALVE	REF. DRAWING
10 TON	48" ID X 118 1/2" L.	STEEL	4500 LBS.	20,000 LBS.	1" SUPERIOR	CKP-19276A
2.5 TON	30" ID X 81 1/2" L.	STEEL	1650 LBS.	5,000 LBS.	1" SUPERIOR	BKP-19274B
MD	12" ID X 53" L.	NICKEL	185 LBS.	430 LBS.	3/4" KEROTEST OR SUPERIOR	CKP-19274A
5 INCH	5" ID X 34-3/4" L.	MONEL	54 LBS.	55 LBS.	3/4" KEROTEST OR SUPERIOR	D-KP 19545A

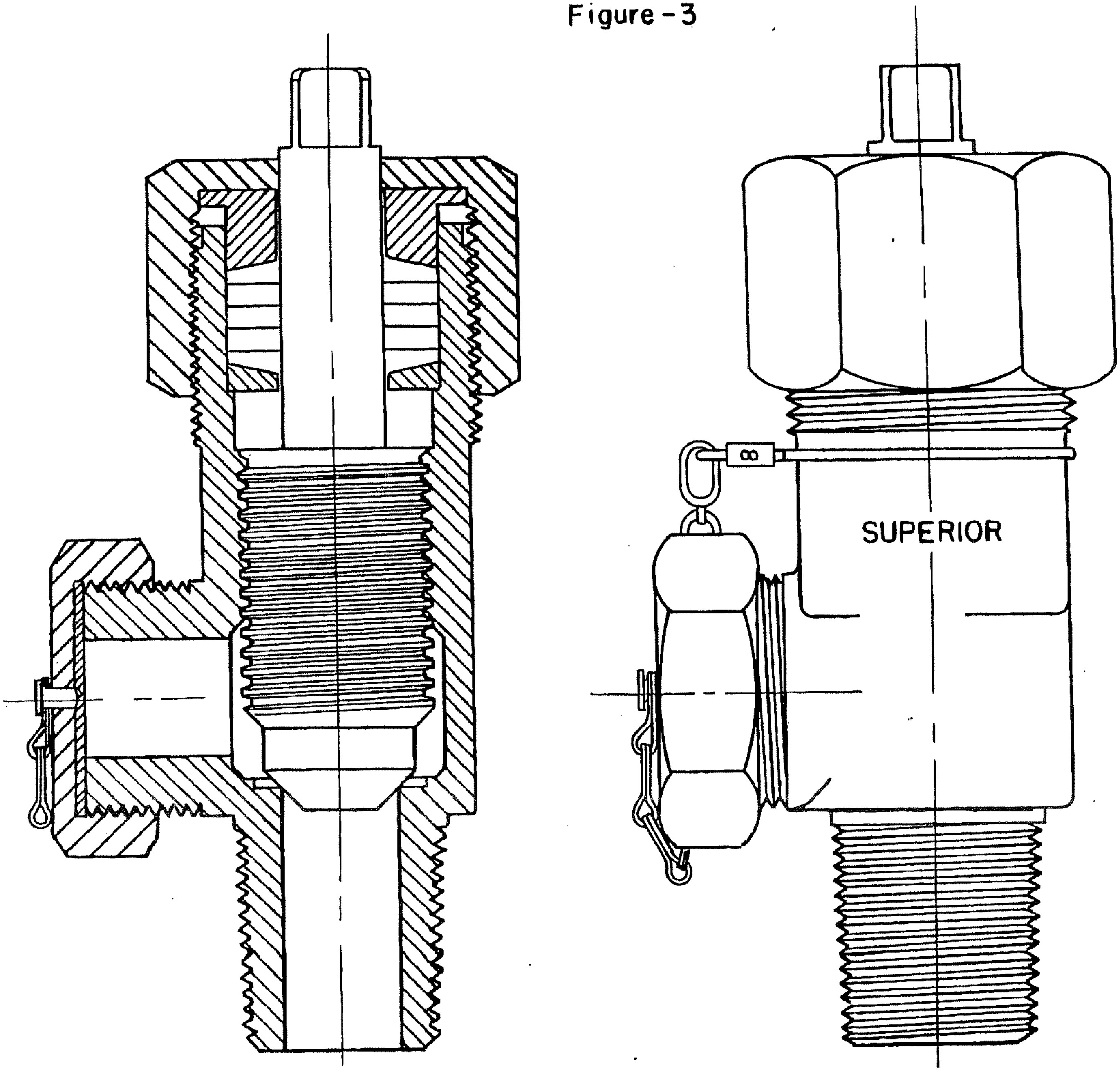
3/4" CYLINDER VALVE

Figure - 2



1" CYLINDER VALVE

Figure - 3



THE UF_6 TRANSFER SYSTEM

The basic elements of a UF_6 transfer system are shown in Figure 4, page 19.

Manifold

The manifold is often made of copper tubing or pipe. More expensive welded monel pipe is sometimes used for permanent installations. The manifold should have a minimum volume consistent with flow requirements.

Pigtail

The pigtail or cylinder adapter, as illustrated in Figure 5, page 20, is used to connect the cylinder to the manifold. Usually, it is made of copper tubing with brass or monel fittings. Teflon gaskets are normally used. Aluminum or copper gaskets may be used, but excessive torque is required to seat them.

Pressure Indicator

The pressure indicator often is a compound pressure gauge with a range of 30 inches Hg to 100 psi. Where remote indication is desired, the use of pneumatic pressure transmitters obviates the necessity for heated lines.

Vacuum System

Two types of vacuum systems are used in handling UF_6. One consists of a positive displacement corrosion-resistant pump capable of obtaining a low suction pressure with a fully fluorinated lubricating oil. The UF_6 passes directly through this system and may be discharged to a UF_6 process system. Another evacuation system, as indicated in Figure 4, page 19, consists of a vacuum pump containing regular hydrocarbon oil preceded by one or more cold traps and a chemical trap in series to remove the UF_6 before entering the pump.

Cold Trap

The cold trap is used to trap UF_6 from the system before opening the system to atmosphere for maintenance, and to collect any UF_6 from the cylinder or system that has to be removed by evacuating the cylinder. The cold traps may be cooled by mechanical refrigeration, dry ice in trichlorethylene, or with liquid nitrogen. The cold traps should have valves on inlet and outlet to facilitate changing. Sometimes baffles are installed in traps to increase thermal transfer.

Chemical Trap

The chemical trap is always used to collect trace quantities of UF_6 which may not have been collected in prior operations. This prevents UF_6 from sludging the vacuum pump oil or from escaping to the atmosphere. Activated alumina of 4 to 8 mesh is often used in the chemical trap. Calcium sulfate is sometimes used instead of alumina in processing medium or highly enriched material. A heavy screen inside the trap, supported just above the inlet, aids flow distribution and prevents the chemical from getting into the inlet line. A flanged top would facilitate removal of the chemical.

Purge Gas Supply

The purge gas is used to sweep out residual wet air or UF_6 and to pressure up systems to locate leaks. A high pressure nitrogen cylinder with a pressure reducing valve should be a satisfactory simple purge gas system. Dry air is also satisfactory. Pressure should be maintained in the purge gas system higher than the pressure in UF_6 system to prevent possible escape of UF_6 into the purge gas system.

Valves

A valve found satisfactory for both liquid and gaseous UF_6 service in the SMMD (Crane Drawing K-4140). This valve has a special forged monel body, socket ends of nominal pipe size, a seating surface machined in the material, a stem sealed by double bellows, and a rising stem which indicates the valve opening. This valve is demountable and has a metal seat.

Similar valves such as SMD (Crane Drawing K-4144) have a special plastic seat and can be used with gaseous UF_6. A small bar stock monel body, plastic seated, bellows sealed stemless valve (HGP-Fulton-Sylphon Drawing 96415) is sometimes used in the 1/4 -inch size for a purge valve.

Globe valves are usually installed in such a manner that the higher pressure is under the valve seat. In cases such as the purge and evacuation valves, where the high pressure side is reversed during the operating cycle, the underside of the seat should be directed toward the UF_6 manifold. When welding the valves to the lines, the valve should be fully open but not back seated. The use of excessive heat in welding will cause distortion of the seat and melt the solder from the bellows. For this reason, it is advisable to disassemble the 1/4-inch, 1/2-inch, and 3/4-inch valves prior to welding; and, as a further precaution, the valve body should be wrapped with asbestos tape and kept wet during the welding operation. The use of sulphur-free asbestos should be used when welding monel to prevent cracking of the metal. After welding, a valve should not be closed until the body has reached room temperature.

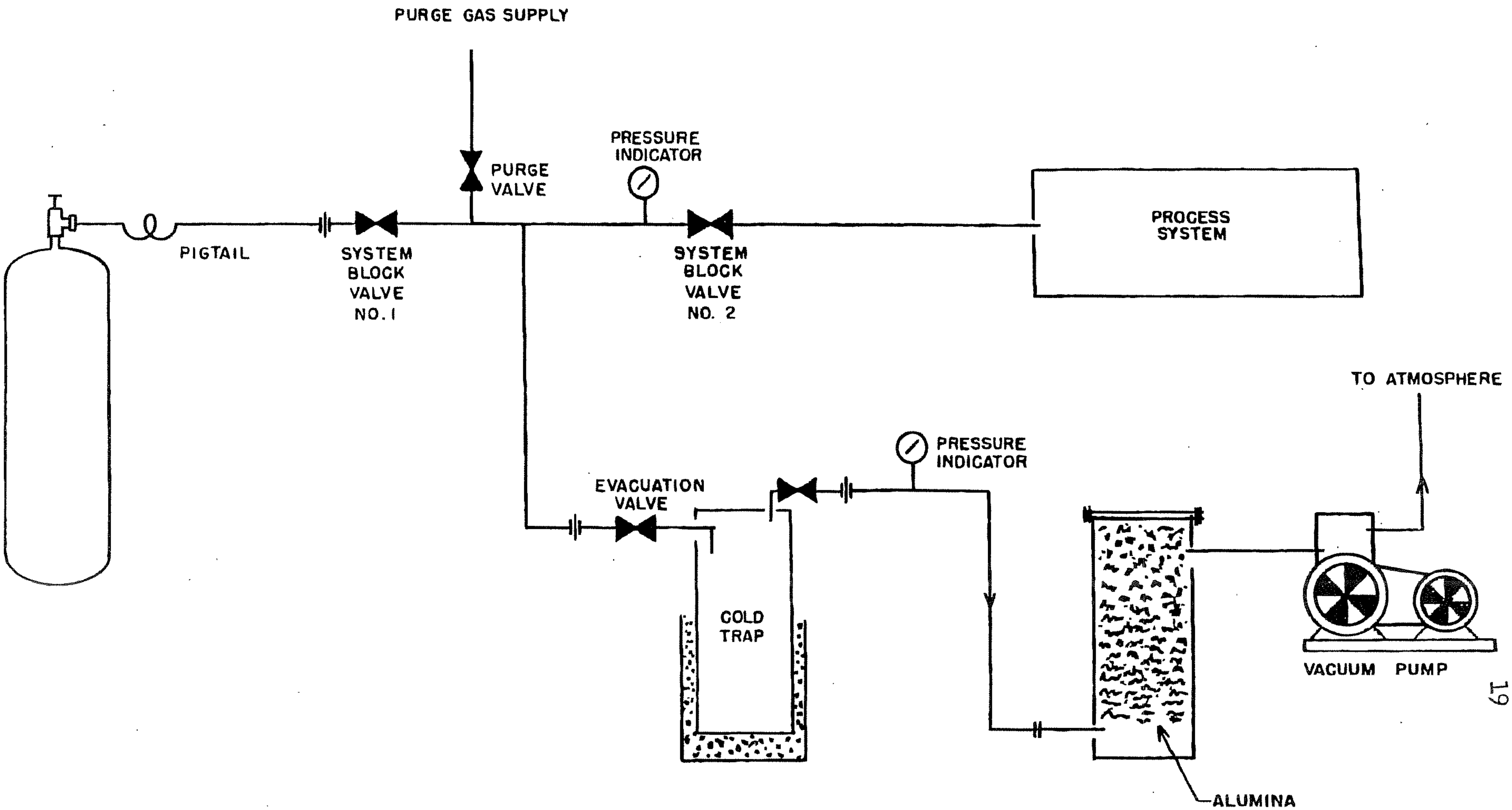
It is recommended that the maximum handwheel torques shown below be used only when necessary since vacuum tightness can normally be obtained at a lesser pressure.

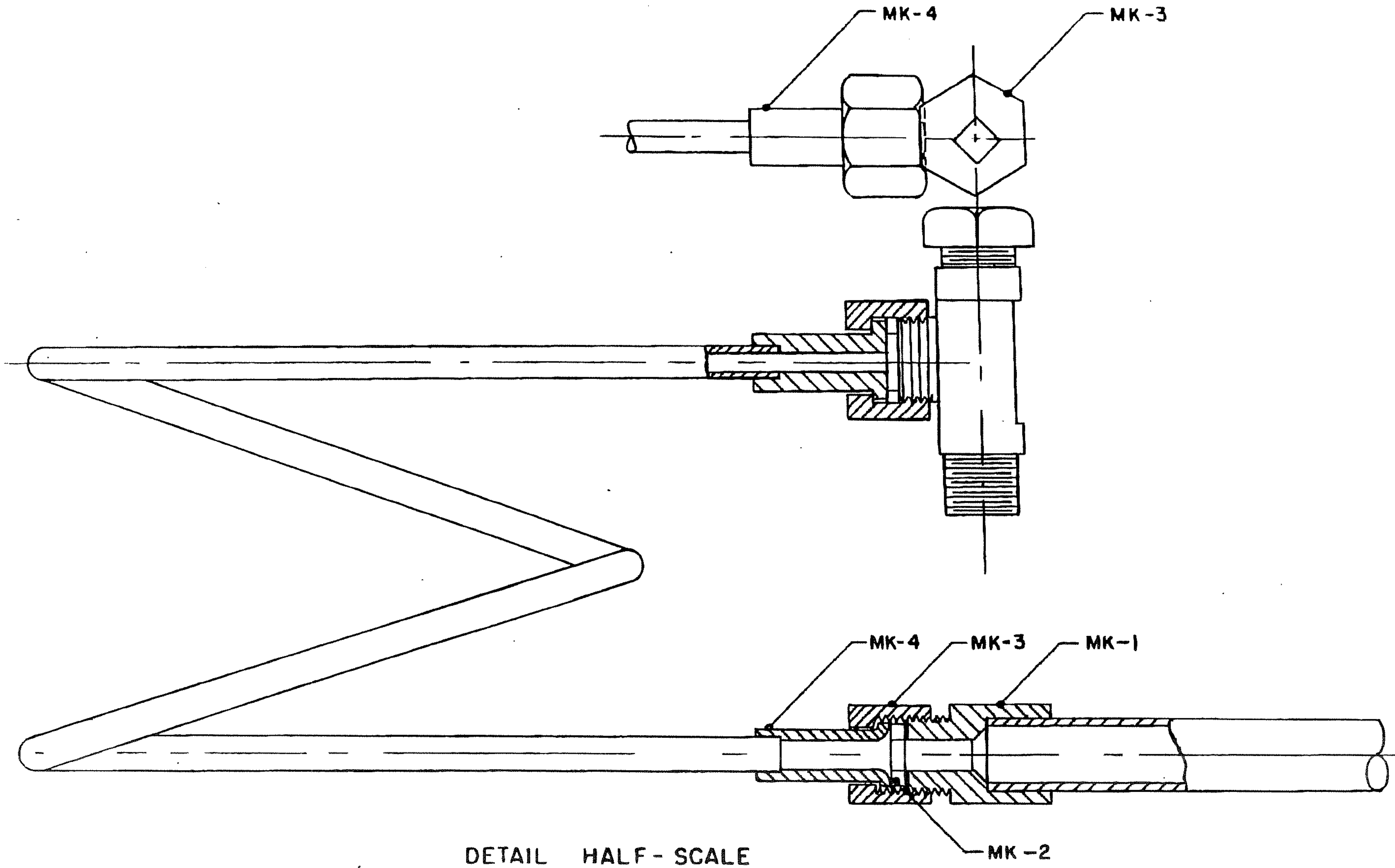
MAXIMUM HANDWHEEL TORQUES IN FOOT POUNDS

Inches	SMMD Valve	SMD Valve
1/4	25	20
1/2	45	20
3/4	57	27
1	68	30
1 1/2	92	40
2	115	50
3	175	75

TYPICAL UF_6 TRANSFER SYSTEM

Figure - 4





DETAIL HALF-SCALE

PIGTAIL

Figure - 5

VAPOR AND LIQUID TRANSFER OF UF_6

Operation of Cylinder Manifold

A manifold system, as depicted in Figure 4, page 19, is used in removing UF_6 from cylinders and in filling cylinders with UF_6 in either the liquid or gaseous state. After connecting the cylinder to the manifold, the connector should be checked for absolute tightness by evacuating and isolating the system and observing for pressure rise. Further checks for a tight system may be warranted depending on the value of the material handled. During a transfer of UF_6 to or from a cylinder, the system block valves and the cylinder valve are open for the material to flow between the cylinder and the system. To disconnect the system, the cylinder valve and the system block valve No. 2 are closed and the manifold purged by alternately evacuating and pressuring until the residual UF_6 concentration is diluted to less than 10 parts per million. The pressure indicator is used to detect possible line restrictions and to monitor the purging operation. The desired UF_6 content can be obtained in five "purges" if the evacuation system is capable of evacuating the system to 5 psia., and the dry air or nitrogen pressure is available at 50 psig.

Condensation in the line increases the difficulty of purging the system, the amount of UF_6 evacuated to the cold trap, and the danger of UF_6 being left in the manifold system at the end of the operation. Any UF_6 left in the system may be released to the atmosphere, thereby contaminating the area, creating a health hazard, and resulting in a loss of UF_6. Uranium hexafluoride, not purged from the system or released to the atmosphere, may be hydrolized by any subsequent introduction of wet air and result in a deposit plugging the manifold or at least restricting the flow. If UF_6 of varying isotopic assays is processed, any material of one assay left in the manifold may mix with UF_6 of another assay processed later resulting in isotopic discrepancies.

Cylinder Filling Technique

Cylinders are filled by solidifying UF_6 from either the gaseous or liquid state inside the cylinder.

Uranium hexafluoride in the gaseous state enters the cylinders through heated lines and valves and is solidified by means of partially submerging the cylinder in a cold solution such as trichlorethylene and dry ice. Progressively submerging the cylinder will permit greater cylinder trapping capacity by preventing the accumulation of solidified UF_6 in the upper portion of the cylinder before the bottom is filled. Cylinder filling by this technique may be hindered by the accumulation of non-condensible contaminants in the cylinders. In that event, the source of UF_6 is valved off and the cylinder and manifold are evacuated to the cold trap for a short time.

In the liquid state UF_6 is drained by gravity into the evacuated cylinder through heated lines and valves where it is solidified at room temperature. The cylinder can be either in the vertical or horizontal position with the valve at the highest point in either case. The temperature of the liquid drain line is controlled at a temperature of approximately 165° F. for a UF_6 pressure of 30 psia. If this temperature drops as low as 150° F. or rises as high as 200° F., there is a possibility of flow restrictions due to solidification or excessive vaporization. The cylinder valve and surrounding area of the cylinder must be heated to prevent the UF_6 from solidifying and thus plugging the cylinder inlet. Heating of the cylinder valve and surrounding area can be accomplished satisfactorily by employing several heat lamps directed on the base of the cylinder valve.

Care should be taken that the cylinder is not overfilled. Uranium hexafluoride undergoes a sizeable change in density as it is liquefied from the solid, and overfilling creates a serious hazard of hydrostatic rupture of the container. If a cylinder is overfilled, the conventional method for reducing the amount of material involves transferring the material by connecting the overfilled container (at room temperature) to a refrigerated cylinder or cold trap and carrying out a low temperature distillation. This is a slow process, but the high pressure release of uranium hexafluoride, resulting from hydrostatic rupture, can be very dangerous.

Cylinder Emptying Technique

The contents of a cylinder may be removed in either a gaseous or liquid state.

The material can be vaporized by placing the cylinder in either a horizontal or a vertical vaporizer using a hot water bath, steam bath or controlled electric heat. Vaporization temperatures should not exceed 300° F. since the specific volume of UF_6 at 300° F. is used to determine maximum net weight of UF_6 permissible in a given cylinder. Heating to temperatures in excess of 300° F. may produce high hydrostatic pressures resulting in cylinder rupture.

In order to determine when vaporization from a cylinder is complete, a check may be made by closing system block valve No. 2 and observing the pressure rise. The absence of a pressure rise indicates that vaporization is essentially complete.

Removing UF6 from cylinders in the liquid state requires that the contents be liquefied before withdrawal and the cylinder placed in such a position that the cylinder valve is below the liquid while withdrawal is taking place.

Regardless of the method of transfer, some residual uranium will remain in the cylinder. This "heel" is comprised of UF_6 vapor and in some cases non-volatile compounds. The residual UF_6 is removed by purging and evacuation through the cold trapping system. If the heel is not removed in cold-trapping, the residue may be either corrosion products or UO_2F_2 which is formed when wet air is inadvertently admitted to the cylinder. If the value of the material warrants, water may be introduced through the cylinder valve and the cylinder washed out. The possibility of evolution of HF should be considered. The cylinder should be dried and evacuated after it is washed out. The following is a more detailed example of a cylinder decontamination procedure used when necessary to thoroughly clean the inside of a cylinder that has two valves:

Circulate hot (180° F.) 5 percent ammonium carbonate aqueous solution through the cylinder until the uranium concentration of the ammonium carbonate solution shows no increase. Drain all ammonium carbonate solution out of the cylinder. Circulate hot (180° F.) water through the cylinder for 30 minutes and then drain the water from the cylinder. The step in the previous sentence is repeated. Blow dry air through the cylinder for 30 minutes. Rinse the cylinder with isopropyl alcohol and dry out with dry air. Evacuate the cylinder to at least 28 inches of mercury. Valve the vacuum pump off and check for a pressure rise due to atmospheric leakage. If there is no leakage, continue to evacuate the cylinder for 6 hours to remove all traces of water and alcohol.

Below is a typical transfer procedure:

Problem: To vaporize 55 pounds of UF_6 from a 5-inch cylinder into the process system. Assume the manifold is properly heated and temperature controlled.

1. Place cylinder in vaporizer.
2. Connect to manifold with clean pigtails and gaskets.
3. Open all valves except cylinder valve, purge valve and valve to process system.
4. Start the vacuum pump and evacuate manifold.
5. Close cold trap inlet valve.
6. Pressure system up to 5 or 10 psig. by opening purge valve and then close purge valve.
7. Open cold trap inlet valve until system is evacuated; then close valve.
8. Observe pressure indicator as required for any significant pressure rise indicating a leak.
9. If a leak is indicated, the system is pressured to 5 psig., and the leak is located by brushing soap solution on possible leak locations such as welds, pigtail connections, etc.
10. After leak is repaired, the system can be pressured up and leak rated.

11. If leak rate is satisfactory, the manifold is evacuated and leak rated at a vacuum.

12. Close valve in line to cold trap.

13. Open cylinder valve. Heat should not be applied to cylinder before this step. Dangerous pressures could be built up if the cylinder is filled above the recommended capacity or contains impurities such as Freon 114.

14. Heat cylinder until pressure in manifold is above process system.

15. Open valve into process system as required.

16. When cylinder is believed to be empty as indicated by weight loss or by pressure drop in the manifold, the valve to the process system is closed. The cylinder is not empty if the pressure continues to rise.

17. If pressure does not rise, the manifold and cylinder are evacuated through cold trap for at least 15 minutes to remove residual quantities (heels) of UF_6 remaining in the cylinder.

18. Close valve to cold trap. Pressure manifold and cylinder to 5 psig. with purge gas. Close purge valve and open valve to cold trap.

19. Repeat step 18 as required to remove all traces of UF_6.

20. Close the cylinder valve and the valve on the manifold to the pigtail.

21. Disconnect the pigtail and remove the cylinder for final weighing.

22. If weight indicates that the cylinder is empty, place the cap on the valve outlet and the valve protector cap on the cylinder which is then ready for reuse.

23. If cylinder weight indicates it is not empty, the cylinder can be reconnected to the pigtail, heated longer or to a higher temperature and evacuated to the cold trap for a longer period.

24. If a reweighing of the cylinder indicates that the heel has not been removed, and if conditions warrant, the above step can be repeated, or the cylinder may be washed.

25. <u>CAUTION</u>: In the operation of the manifold system, never shut down the vacuump pump against a vacuum in the manifold unless pump suction valve is closed. Oil may be pulled from the pump and through the chemical trap which could react explosively with UF_6.